



Non-Destructive Testing of Metal Backed Materials Using Reflection Mode Terahertz Spectroscopy

Surya Prakash Singh^{*(1), (2)}, Nilesh Kumar Tiwari⁽¹⁾, and M. Jaleel Akhtar⁽¹⁾

(1) Indian Institute of Technology Kanpur, Uttar Pradesh, India, 208016

(2) International Institute of Information Technology Bhubaneswar, Odisha, India, 751003

Abstract

In this paper, we propose and demonstrate an accurate, fast and thickness independent reflection mode terahertz spectroscopy technique to determine the material parameters of the test specimen. A generalized closed form expression is derived to extract the index of refraction and thickness of the material under test. The proposed method removes the most influential error introduced in the determined index of refraction because of the mechanical measurement of the sample thickness. The method is numerically tested for two configurations, viz. metal backed specimen and air backed specimen. The extracted material parameters for both configurations are in good agreement with the reference data. The proposed technique is fast, non-destructive and self-calibrated; therefore it can find applications in automobile and aviation industries, where the materials are tested with metal at the backside of the specimen.

1. Introduction

The terahertz (THz) time domain spectroscopy (TDS) has become a conventional technique to characterize the materials in the THz frequency range for the last two decades. This technique is able to characterize the test specimen over a wide band frequency band ranging from 100 GHz to several THz. The THz-TDS approach can be broadly classified into two categories, viz. the transmission mode THz-TDS, and the reflection mode THz-TDS. Usually, the transmission mode spectroscopy is applied to characterize the low loss materials and reflection mode spectroscopy is used to characterize the high lossy and absorbing liquid materials. In transmission mode, the waveform received at the transmission end after interaction with the sample material (called as sample measurement) is compared with the waveform obtained without sample material (called as reference measurement), to obtain the material parameters [1], [2]. In most of the earlier proposed techniques [3], [4], the complex index of refraction and the thickness of the material under test (MUT) is simultaneously obtained with the help of optimization process. The optimization based techniques [3], [4] may not be suitable for real time material characterization applications as they may take time to determine the material parameters. The methods without employing any optimization process appear to be more appropriate for real time monitoring applications

[5], [6]. In literature, some other alternative methods such as multiple angle method [7] and ellipsometry measurement method [8] have also been proposed to determine the material properties. Both proposed methods in [7], [8] require multiple measurements at different incident angles, which may not be possible for some practical cases. While in reflection mode spectroscopy, the liquid sample is placed at the top of the quartz or other types of window materials and the ratio between the reflected signals from the interface between the window material and liquid sample to the interface between air and the window material, forms the basis of extracting algorithms [9]-[12]. Unlike the transmission mode TDS, the reflection mode TDS does not require the access of the other side of MUT, therefore the reflection mode TDS also finds applications in the testing of those specimen in which the other side of the specimen cannot be accessed. The potential applications of reflection mode TDS are the thickness determination of paint layer in automobile industry, determination of the properties of plastic materials used in aviation industry and screening of goods at security checkpoints [13].

The proposed technique in this article uses the first three reflected pulses from MUT interfaces, caused by Fabry-Perot effect, to determine the material parameters. The proposed method is fast and non-invasive which suits quite appropriately for the industrial applications such as real time material characterization system and security screening application. It also removes the most influential error introduced in the determined refractive index because of the incorrect measurement of the sample thickness. The index of refraction in the present situation is actually determined from the measured reflected signals only without requiring any prior knowledge of the sample thickness. The proposed approach also facilitates the determination of the sample thickness in terms of the extracted refractive index of the material under test. The applicability of the proposed method is numerically tested in the THz frequency range for different samples.

2. Model to Extract the Material Parameters

Fig. 1 shows the measurement configuration for the normal-incidence reflection TDS and a simple illustration of the measured reflected signals are shown in Fig. 2. The multiple signals collected at the reference plane BB' are offset in the Fig. 2 for clear visualization, as for the

normal incidence configuration the multiple reflected signals and the input signal will overlies each other. A THz source and receiver are assumed to be kept at AA' and BB' reference planes, respectively which transmit and receive the signals. The sample of thickness L is kept at a distance L_1 and L_2 from the reference plane AA' and BB' , respectively. The receiver receives the multiple signals due to Fabry-Perot effect, however, the amplitude of the first few signals would have only significant amplitude level. These multiple time domain signals are separated with time gating window function, afterwards FFT is applied to each separated time pulse. The frequency domain equation for the first, second and third multiple signals received at BB' reference plane can be written as

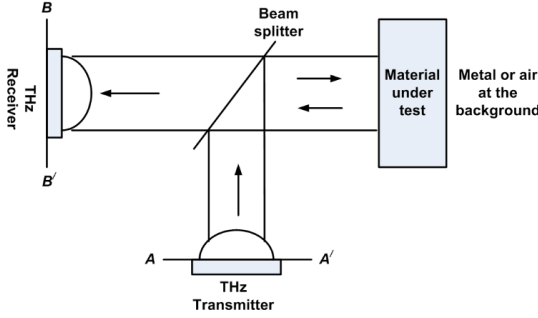


Figure 1. Measurement configuration for the normal-incidence reflection TDS.

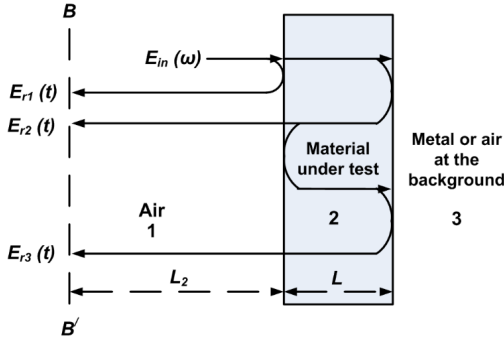


Figure 2. Fabry-Perot model depicting multiple reflected signals from a planar homogenous sample.

$$E_{in}(\omega) = B_1 E_s(\omega) \exp(-j\omega n_{air} L_1/c) \quad (1)$$

$$E_{r1}(\omega) = r_{12} B_2 E_{in}(\omega) \exp(-j\omega n_{air} L_2/c) \quad (2)$$

$$E_{r2}(\omega) = t_{12} r_{23} t_{21} B_2 E_{in}(\omega) \exp(-j\omega n_{air} L_2/c) \times \exp(-j\omega n_{sam} 2L/c) \quad (3)$$

$$E_{r3}(\omega) = t_{12} r_{23} r_{21} r_{23} t_{21} B_2 E_{in}(\omega) \exp(-j\omega n_{air} L_2/c) \times \exp(-j\omega n_{sam} 4L/c) \quad (4)$$

where, n_{air} and n_{sam} represent the complex index of refraction of air and sample, respectively, $E_s(\omega)$ is the transmitting signal at reference plane AA' , $E_{in}(\omega)$ is the incident signal at the Air-MUT interface plane, B_1 and B_2 are the beam splitter coefficients for the incoming signals from the THz transmitter and reflected signals from the MUT interfaces, respectively, ω is the angular frequency, c corresponds to velocity of light in vacuum, r and t

denote the Fresnel reflection and transmission coefficients for normal incidence, respectively.

The Fresnel transmission and reflection coefficients for normal incidence at i - j interface, can be defined in terms of the complex refractive index of i^{th} and j^{th} layer, as

$$r_{ij} = \frac{n_i - n_j}{n_i + n_j} \quad (5)$$

$$t_{ij} = \frac{2n_i}{n_i + n_j} \quad (6)$$

From (2), (3) and (4), we get

$$\frac{E_{r2}(\omega)}{E_{r1}(\omega)} \times \frac{E_{r2}(\omega)}{E_{r3}(\omega)} = \frac{t_{12} t_{21}}{r_{12} r_{21}} \quad (7)$$

It can be observed that (7) does not contain the reflection coefficient r_{23} corresponding to the MUT and Background material interface, therefore, (7) is valid for all kind of semi-infinite background materials. On substituting Fresnel reflection and transmission coefficients from (5) and (6) into (7), we obtain

$$\frac{E_{r2}(\omega)}{E_{r1}(\omega)} \times \frac{E_{r2}(\omega)}{E_{r3}(\omega)} = \frac{4n_{air} n_{sam}}{(n_{sam} - n_{air})(n_{air} - n_{sam})} \equiv X \quad (8)$$

By putting $n_{air} = 1 - j0$ for simplification, (8) reduces to

$$n_{sam}^2 X - n_{sam} (2X - 4) + X = 0 \quad (9)$$

On solving (9) for n_{sam} , we get

$$n_{sam} = n_{real} - jn_{img} = \frac{(X - 2) \pm \sqrt{(X - 2)^2 - X^2}}{X} \quad (10)$$

The correct root of equation (10) is chosen on the basis of sign of n_{img} . The value of n_{img} obtained from (10) should be positive as the negative sign has been already taken care in the expression. Equation (10) is a closed form expression to calculate the complex index of refraction. The determined n_{sam} depends only on the measured multiple signals data in frequency domain, thus removing the error associated with the sample thickness measurement. It also alleviates the requirement of reference signal measurement, since it is a self-calibrating technique.

The sample thickness in the present situation can be determined from the time delay measurement between the successive multiple reflected signals. If the first reflected signal, $E_{r1}(t)$, is received at the time t_1 and the next successive reflected signal, $E_{r2}(t)$, is received at the time t_2 , then from Fig. 2, the equations for t_1 and t_2 can be written as

$$t_1 = \frac{n_{air} L_1}{c} + \frac{n_{air} L_2}{c} \quad (11)$$

$$t_2 = \frac{n_{air} L_1}{c} + \frac{n_{air} L_2}{c} + \frac{2n_{real} L}{c} \quad (12)$$

From (11) and (12), we get

$$L = \frac{c}{n_{real}} \times \frac{t_2 - t_1}{2} \quad (13)$$

which basically represents the required equation for estimating the sample thickness in terms of the retrieved refractive index of the test specimen..

3. Numerical Validation of the Proposed Technique

The validation of the proposed reflection mode TDS algorithm is done numerically using a 3D full wave Electromagnetic Simulator, Computer Simulation Technology Microwave Studio (CST-MWS). Fig. 3 shows the simulation geometry, where a sample with metal background is kept at a distance L_2 from the receiving port. The boundary conditions are applied to ensure the plane wave propagation and the transient domain solver is used to study the transient behaviour of the system. The technique is tested for two configurations, viz. metal backed MUT and air backed MUT. In numerical simulation, four type of samples are taken, viz. low loss samples (HDPE, Quartz), medium lossy sample (Pyrex). The parameters provided for simulation are tabulated in Table 1. The received multiple time domain signals for different samples are depicted in Fig. 4 and Fig. 5. These multiple time domain signals are separated with time gating window function, afterwards IFFT is applied to each separated time pulse. The obtained frequency domain data corresponding to the first, second and third multiple signals are supplied to the proposed THz algorithm to extract the complex index of refraction and thickness of samples listed in Table 1.

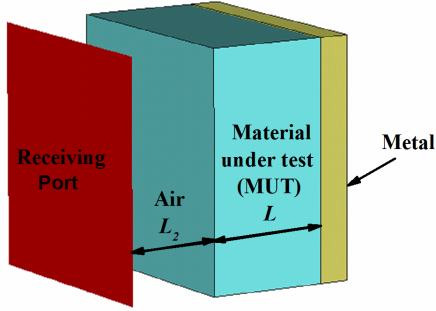


Figure 3. CST simulation model of material under test.

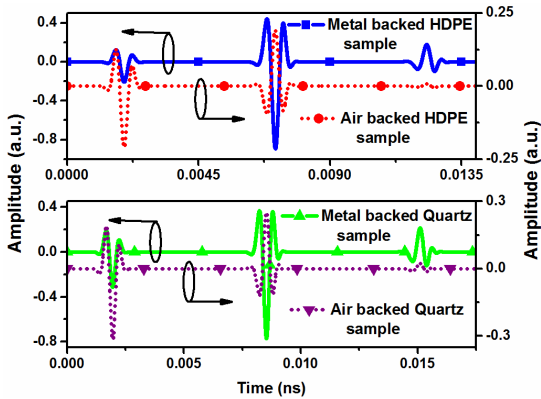


Figure 4. Multiple received signals for HDPE and Quartz samples.

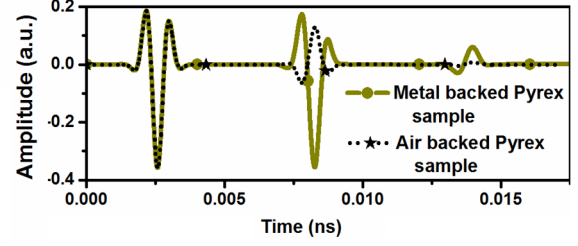


Figure 5. Multiple received signals for Pyrex sample.

It can be observed from Fig. 4 and Fig. 5 that the second and third reflected signals are weak for air background configuration in comparison to the metal background configuration. The amplitude of the reflection coefficient for material-metal interface is always equal to 1, however for other background materials like air, the amplitude of the reflection coefficient for material-air interface depend on the refractive index of the MUT, therefore the amplitude of the second and third received signals have low amplitude for air background configuration.

Table 1 Material Parameters Taken for THz Simulation

Sample	Index of Refraction	Thickness (μm)
HDPE	1.54-j0.0015 [14]	500
Quartz	1.96-j0.0039 [14]	500
Pyrex	2.12-j0.055 [14]	400

Fig. 6 shows the extracted real and imaginary part of the index of refraction for HDPE and Quartz samples. Similarly, Fig. 7 show the extracted index of refraction for Pyrex.

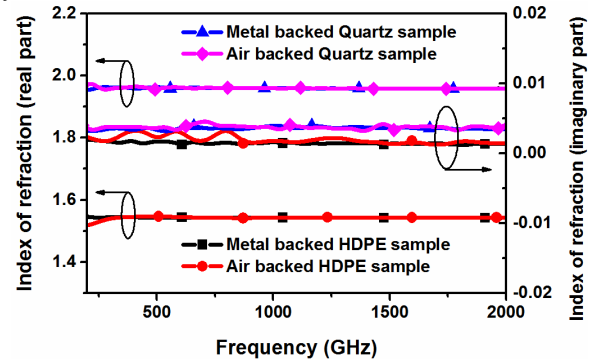


Figure 6. Extracted index of refraction of HDPE and Quartz samples for both configurations.

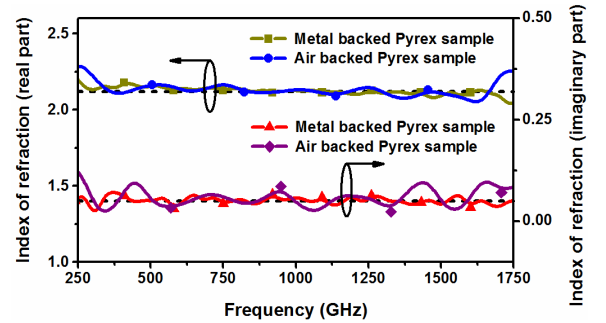


Figure 7. Extracted index of refraction of Pyrex sample for both configurations.

In all of the above figures corresponding to the extracted optical parameters, the dotted line corresponds to the actual data of sample taken during simulation and the continuous line corresponds to the extracted value using the proposed reflection mode TDS algorithm. From the obtained results for HDPE and Quartz samples, it can be seen that extracted real part of the refractive index is highly accurate for both the configurations, however the imaginary part of refractive index for HDPE is more accurate for the metal backed configuration than the air backed configuration. Similarly, it can be observed from Fig. 7 that the extracted refractive index for Pyrex is having good accuracy for metal backed configuration while it shows poor accuracy for air background configuration. The reason behind the error in extracted parameters for air background HDPE and Pyrex samples is the very low amplitude value of the third received pulse. It can be seen from (8) that $E_{r3}(\omega)$ is in the denominator part, and if it is getting very low, the whole expression can shoot up and provide less accurate results. Thus the proposed algorithm is best suited for metal backed configuration and it can also provide results with good accuracy for air backed configuration if the amplitude of the third pulse is not having very low amplitude. The thickness of the samples can be determined using (13).

4 Conclusion

A fast, non-iterative reflection mode time domain technique has been presented to determine the electrical parameters and material thickness of the test specimen. The method is applicable for terahertz frequency band as well as for lower mm-wave frequency band. The proposed approach has been numerically tested in the terahertz frequency range, while it is experimentally validated in the millimetre frequency band. The numerical and measurement results are in close agreement with the actual values taken for the simulation and the data available in literature. The proposed method is quiet accurate, non-invasive and fast enough to be used for real time material characterization and imaging systems. The limitations and possible sources of error of the proposed method have been discussed. The proposed analytical approach can be combined with the optimization process to obtain more accurate results especially for lossy samples.

5. References

1. W F Pawhier, D D Honijk, M Mandell and M N Afsar, "A new method for the determination of complex refractive index spectra of transparent solids in the far-infrared spectral region: Results of pure silicon and

- crystal quartz," J Phys D Appl Phys, **10**, 1977, pp. 509-517.
2. Lionel Duvillaret, Frederic Garet, and Jean-Louis Coutaz, "A reliable method for extraction of material parameters in terahertz time-domain spectroscopy," IEEE J Sel Top Quantum Electron, **2**, 1996, pp. 739-746.
3. T. D. Dorney, R. G. Baraniuk, and D. M. Mittleman, "Material parameter estimation with terahertz time-domain spectroscopy," J Opt Soc Am A, **18**, 2001, pp. 1562-1571.
4. Ioachim Pupeza, Rafal Wilk, and Martin Koch, "Highly accurate optical material parameter determination with THz time-domain spectroscopy," Opt Express, **15**, 2007, pp. 4335-4350.
5. F. Huang, J.F. Federici, and D. Gary, "Determining thickness independently from optical constants by use of ultrafast light," Opt Lett, **29**, 2004, pp. 2435-2437.
6. Surya Prakash Singh, Zubair Akhter, and M. J. Akhtar, "Calibration independent estimation of optical constants using terahertz time-domain spectroscopy," Microw Opt Technol Lett, **57**, 2015, pp. 1861-1864.
7. Jose A. Hejase, Edward J. Rothwell, and Premjeet Chahal, "A multiple angle method for THz time domain material characterization," IEEE Trans Terahertz Sci Technol, **3**, 2013, pp. 656-665.
8. Naoki Matsumoto, Tadasu Hosokura, Takeshi Nagashima, and Masanori Hangyo, "Measurement of the dielectric constant of thin films by terahertz time-domain spectroscopic ellipsometry," Opt Lett, **36**, 2011, pp. 265-267.
9. Peter Uhd Jepsen and Bernd M. Fischer, "Dynamic range in terahertz time-domain transmission and reflection spectroscopy," Opt Lett, **30**, 2005, pp. 29-31.
10. Shengyang Huang, et al., "Improved sample characterization in terahertz reflection imaging and spectroscopy," Opt Express, **17**, 2009, pp. 3848-3854.
11. Uffe Møller, David G. Cooke, Koichiro Tanaka, and Peter Uhd Jepsen, "Terahertz reflection spectroscopy of Debye relaxation in polar liquids," J Opt Soc Am B, **26** 2009, pp. A113 A125.
12. Amin Soltani, et al., "Attenuated total reflection terahertz time-domain spectroscopy: uncertainty analysis and reduction scheme," IEEE Trans Terahertz Sci Technol, **6**, 2016, pp. 32-39.
13. Ke Su, Yao-Chun Shen, and J. Axel Zeitler, "Terahertz sensor for noncontact thickness and quality measurement of automobile paints of varying complexity," IEEE Trans Terahertz Sci Technol, **4**, 2014, pp. 432-439.
14. Jose A. Hejase, Pavel R. Paladhi, and Premjeet Chahal, "Terahertz characterization of dielectric substrates for component design and nondestructive evaluation of packages," IEEE Trans Terahertz Sci Technol, **1**, 2011, pp. 1685-1694.